# MEETING THE CHALLENGE:

## Activating Through Holes in Teflon® Material

When processing Teflon® panels through plating, your manufacturing process might have more flexibility than you think.

By **Lou Fierro**

It is very difficult to plate to Polytetrafluoroethylene (PTFE) material. Its chemical hydrophobic properties, together with low surface energy, cause the surface to repel liquids making it very difficult to plate. To successfully plate PTFE material, it must be treated prior to applying the seed layer for plating.

There are several pre-electrolytic processes commonly used in the industry; hereinafter referred to as copper deposition (Cu Dep). The two common methods of treating Teflon® material are Sodium etch (Na) chemical treatment, and a gas plasma process using a combination of $H_2$ (Hydrogen) and $N_2$ (Nitrogen). Each method successfully activates (renders) hole walls for metalizing. However, since PTFE material has a characteristic that causes it to recover to the untreated condition, plating must be completed before recovery occurs.

This article is based on a Design of Experiment (DOE) which compares the ability of three different plasma processes and Sodium etch chemical processing to activate Teflon® material, and evaluates plating adhesion in relationship to the time a panel is held before Cu Dep.

### Teflon® Material in the Printed Circuit Industry

The uses for Teflon® material extend beyond the microwave applications of past years. For example, board designers now incorporate Teflon® material in the design of voltage-controlled oscillators, IF amplifiers, ground spectrum devices, and other strip-line and micro-strip circuits. In addition, Teflon® is now commonly specified as the required material for fabrication of single, double, and even multilayer panels.

The wider use of Teflon® material is due to the varieties now available with improved specifications: fillers that provide electrical and mechanical stability, a low CTE, low dielectric constant, and low moisture absorption. However, Teflon® material's hydrophobic characteristics present manufacturing challenges as liquids bead and roll off their surface. As a result, Teflon® material requires treatment after drill for both single and double-sided panels. Unless panels are treated prior to Cu Dep, there is little success in covering a hole wall.

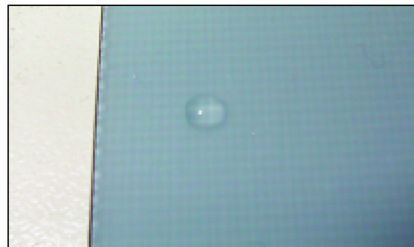

**Figure 1a.** Before Treatment (Hydrophobic).

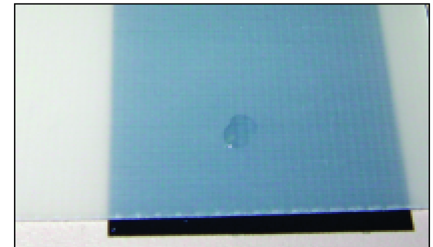

**Figure 1b.** After Treatment (Hydrophilic).

### Standard Methods of Treating Teflon® Material

Teflon® material requires treatment to activate the surface and increase wettability of hole walls and/or board edges. Increased wettability is a change in the material surface tension from hydrophobic to hydrophilic, or from repelling to attracting a liquid. This change is usually evaluated by measuring the contact angle, the angle formed by a droplet of water in contact with the material surface (Fig. 1).

The two most widely used methods of treating Teflon® material are wet chemical processing with Sodium etch (Na) and gas plasma processing using a combination of $H_2$ (Hydrogen) and $N_2$ (Nitrogen). Both methods are successful in lowering the contact angle and sufficiently wetting the material surface required for good metalizing coverage.

Sodium etch poses environmental concerns and safety issues with regard to handling and waste disposal. Plasma with hydrogen has posed concerns regarding its flammability, however proper control eliminates risk (e.g., use of a hydrogen generator—a localized source of hydrogen from the electrolysis of water).

### Activation: Comparison of Sodium and Plasma

To determine the actual effect Na and plasma have on activating Teflon® material, a DOE was developed to compare wettability and its relationship to plating

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adhesion. Both chemical Na and plasma $H_2/N_2$ have a good track record for treating Teflon® material, but there is limited data comparing each process to plating adhesion. Two additional plasma gases were also evaluated, 100-percent He and 100-percent $N_2$, to validate plasma processes without hydrogen.

The DOE was designed to test expectations that 1) without pre-treatment, adhesion will not be possible, and 2) pre-treatment promotes adhesion, but plating capability will fall off over time as the material surface relaxes to pre-treatment conditions.

### Material Characteristics

As stated earlier, Teflon® material is hydrophobic in nature. It also has memory, which means that its surface will eventually return to a non-wettable state after treatment. The length of time it takes for Teflon® material to return to its non-wettable state is called recovery time, typically believed to be 48 hours after treatment. Most manufacturers plate Teflon® material within 24 hours since adhesion degradation is expected after 48 hours.

The two Teflon® materials selected for the study were Taconic woven glass-reinforced PTFE with different ZCTE and dielectric constants. Both materials are commonly used in Teflon® board manufacturing and have non-wettable characteristics. All 32 panels (16 of each material) were a standard size of 12 x 18 x 0.062 in. with 0.020-in. diameter holes (average aspect ratio for today's Teflon® PCBs). The panels were unclad in order to collect plating adhesion data, a measurement that could not have been made in plated through holes. All plasma process conditions were held constant (i.e., power, pressure, frequency and time) except for the change in process gas.

### Collecting the Data

To gather data, the project included measurement of treatment effectiveness as a function of time in order to establish the process window. All panels for both materials were processed on the same day. A four-day holding period was selected based on the maximum time a board manufacturer might hold panels before plating. The panels were plated with formaldehyde electroless copper followed by an acid-based electroplate copper with an average thickness of 0.0007 inches. On four consecutive days, panels representing the four processes were released for plating. Contact angles and surface energy were recorded each day. This data served two purposes: to measure recovery time (i.e., return to the non-wettable state) and to measure their relationship to plating adhesion.

After plating, each panel was evaluated by back lighting, tape test, peel strength and solder float. The data was recorded on each of the four days, and results were compared to the contact angle and surface energy.

## RESULTS

### Contact Angle

Contact angle and surface energy were measured prior to treatment in order to establish a baseline for both materials. The contact angles and surface energy were very high, 100- and 105 degrees. These measurements were expected to be in the non-wettable range, yielding poor Cu Dep and adhesion.

As expected, the untreated sample panels processed through electroless failed. As a result, the remaining tests (i.e., tape test, peel strength, and solder float) could not be performed on the untreated samples. Their contact angle and surface energy measurements established the non-platable conditions of each material type and were the baseline measurements to which each process was compared over the four days.

Sodium etch had the least fluctuation in contact angles with relationship to recovery over the four-day period, ranging from 59 to 67. Of the three plasma process, the $H_2/N_2$ process showed the lowest contact angle of 23 on Day 1. The other two plasma processes, He and $N_2$, did not lower the contact angle as much as Na and $H_2/N_2$. However, both processes were considered in the wettable range and sufficient for Cu Dep. Contact angles ranged from an average of 60 to 103 for the three plasma processes.

Over the four days, the panels treated with Na recovered the least and remained in the wettable range. The plasma-treated materials continued to recover each day, eventually returning to their pre-treated condition after Day 4. Plating failures were anticipated as the material surface relaxed to pre-treatment conditions.

### Surface Energy

In addition to contact angle, surface energy is also used to establish material wettability. Surface energy is a measure of surface tension and its ability to attract or repel a liquid. Low surface energy readings indicate a non-wettable surface. Both untreated material types had surface energy measurements less than 30 dynes, indicating a poor surface for plating which was confirmed when an attempt to plate to them failed.

After treatment, all four processes had surface energy measurements greater than 60 dyne. These readings were considerably higher than the pre-treated panels and suitable for good wetting of Cu Dep. Measurements recorded over the four-day holding period demonstrated that the Na etch and plasma $H_2/N_2$ processes held at greater than 60 dyne with no change from the first day and were still in a wettable state. He and $N_2$ plasma processes continued to lower over the four-day period and were slightly better than the pre-treated condition, indicating potentially poor plating capability.

### Plating Adhesion Results

**Backlighting.** The first test was to determine how successful coverage was over the four consecutive days for each of the four processes. Panels were backlit with a black light and plated holes were visually inspected for voids. Since the panels were unclad prior to plating, each panel could be evaluated over a much larger surface area than normally available on Teflon®-printed circuit panels. All panels passed: voids were not observed in the plated through holes, and outer surfaces had complete coverage.

Contact angle measurements and surface energy for the Na etch and plasma $H_2/N_2$ were as good as expected. Although panels processed with He and $N_2$ plasma recovered over the four-day period, they too showed good coverage.

**Peel Strength.** After the panels were reviewed for plating coverage and voids, samples representing each process were subjected to a tape test and adhesion peel strength. Failures were predicted over the four-day period that would require cross-referencing back to contact angle. However, voids were not found from any of the processes, so the data was used only as a comparison.

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A tape pull test, per specification IPC-TM-650 2.4.1, was performed on each of the panels. As previously noted, the area of Teflon® material requiring activation was far greater than would ordinarily be found in the drilled through holes of a panel. In spite of this, there was no evidence of copper on the tape or peeling from the panels. All processes were successful over the four-day holding period.

Following the tape test, actual peel strength data was recorded. The peel strength was measured by applying a load at a constant rate of 1.2 lbs/minute. Overall, the Na etch process had slightly higher readings than the plasma processes, yet there was very little change in peel strength on Day 4 for any of the processes. This peel strength data demonstrated that as the materials began to recover after four days, there was still sufficient wettability to allow good adhesion, even on the large surface area of unclad panels.

The tape and peel strength test results were acceptable, yet poor adhesion was expected as the material recovered to pre-treatment conditions. Additional testing is required to determine the reasons why this occurred.

**Solder Float.** The solder float test was the most stringent and critical of all tests for plating adhesion. Solder float is the method most commonly used by PCB manufacturers to determine the reliability of a plated through hole. Coupons representing each process (i.e., Na etch, plasma $H_2/N_2$, He, and $N_2$) and each of the four days were subjected to a 550°F solder float for 10 seconds. Microsections were prepared and evaluated at 400x for plating adhesion. Day 4 sections looked as good as Day 1, indicating that all four processes for both material types were successful. The results of this thermal stress test indicated that the hole walls were sufficiently activated and wettable to ensure reliable plating, even for the processes where recovery had occurred. (Refer to Fig. 2 for cross-section photos.)

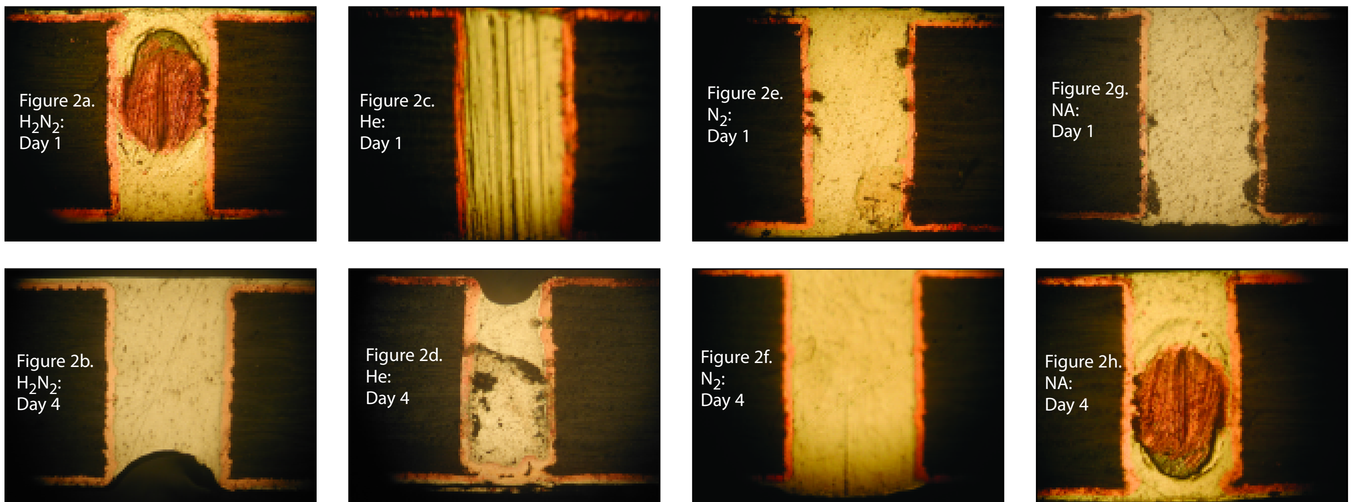


**Figure 2.** Cross-Sections Following Solder Float.

## Summary

The purpose of this experiment was to evaluate four processes used to activate PTFE and to determine the process flexibility a board manufacturer has in preparing Teflon® panels for Cu Dep.

Summarizing the four processes, plasma $H_2/N_2$ had the lowest contact angle on Day 1. However, all the plasma processes continued to recover over the four-day holding period. Sodium etch had the least change in relationship to contact angle and wettability. Based on the data collected from the plating adhesion tests, all processes had sufficiently activated the Teflon® material regardless of recovery. The good results from the familiar Na and plasma $H_2/N_2$ processes were expected.

This study validated two additional processes that can be used for activating Teflon® material when there are concerns with using Na and $H_2/N_2$. Although recovery was seen from Day 1 to Day 4, thermal stress results show that once the contact angle and surface tension changed, plating was successful.

PCB manufacturers can choose a chemical process or one of three plasma processes to activate Teflon® panels. For all processes, panels can be held four days before Cu Dep.

Typically, contact angle and surface energy measurements are used to indicate the success of plating adhesion. Plating adhesion was expected to fail over the four days as the material surface relaxed to pre-treatment conditions. Surprisingly, although contact angle measurements increased over time, there were no statistically significant changes in adhesion. Both peel strength and solder float test results on Day 4 were as successful as those on Day 1.

Although wettability is a good measure of a successful pre-treatment process, further testing is required to determine the ultimate recovery time and the mechanism that is enhancing the adhesion. The DOE raised questions that require further study. To be continued…

**Author's note:** This study was based on two different types of reinforced PTFE material; other constructions may not have the same results and could require adjustments to process parameters. **CT**

**References**

1. L. Fierro, "PTFE Wettability for Electroless Copper and Direct Metalization," IPC Printed Circuits Expo 2004.

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